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IS 1448-127 (1988): Methods of test for petroleum and its products, Part 127: Determination of iron in petroleum coke [PCD 1: Methods of Measurement and Test for Petroleum, Petroleum Products and Lubricants]

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Indian Standard

(Reaffirmed 2006)

METHODS OF TEST FOR PETROLEUM AND ITS PRODUCTS

[P : 127]

DETERMINATION OF IRON IN PETROLEUM COKE

(First Reprint AUGUST 1997)

UDC 665.777.4 : 543 [546.72]

1. SCOPE

1.1 This standard describes an analytical test procedure for the determination of iron in petroleum coke.

2. APPARATUS

2.1 Spectrophotometer or Colorimeter — With 1 cm absorption cell and suitable filters.

2.2 Platinum Crucible with Cover — 30-ml capacity.

2.3 Assorted Volumetric Flasks, Pipettes, Beakers

3. REAGENTS

3.1 Hydroxylamine Hydrochloride ($NH_2OH.HCl$) (10 Percent Solution) — Into a 250-ml beaker, weigh 25 g $NH_2OH.HCl$, add about 100 ml water and stir until dissolved. Transfer to a 250-ml flask and dilute with water to mark. Mix well. Store in a refrigerator and discard if any colour develops.

3.2 2, 4-Dinitrophenol Indicator (0·1 Percent Solution) — Dissolve 0·5 g of 2,4 dinitrophenol *p*H indicater in water and dilute to 500 ml with water.

3.3 1, 10 Phenanthroline (1·5 Percent Alcoholic Solution) — Into a 100 ml beaker, weigh 1·5 g of 0-phenanthroline monohydrate, add 95 percent ethanol and stir until dissolved. Transfer to 100-ml graduated flask and dilute to the mark with ethanol. Store in a refrigerator and discard if any colour develops.

3.4 Hydrocholric Acid (6 N Solution) — Dilute 12 N concentrated hydrochloric acid with an equal volume of water. Dispense in a dropper bottle.

3.5 Ammonium Hydroxide (NH_4OH) (6 N Solution) — To 120 ml of distilled water, add 80 ml of concentrated ammonium hydroxide (15 N), dilute to 200 ml in a volumetric flask. Mix well and store in a polyethylene bottle.

3.6 Bromine Water — Add 10 ml bromine to 1 litre of water. Mix and allow to stand for 24 h.

4. PROCEDURE

4.1 Preparation of Standard Iron Solution (1 ml = 0·1 mg Fe) — Dissolve 0·103 2 g of pure (99·9 percent) iron wire in 6 N HCl. Add about 5 ml of bromine water solution and boil off to remove excess bromine. Cool, transfer to a one-litre volumetric flask, dilute to mark and mix well. This solution may be used as a 'primary standard' or it may be standardized in the usual manner volumetrically against a standard potassium dichromate solution.

4.2 Preparation of Standard Iron Solution (1 ml = 0·01 mg Fe) — Dilute 25 ml of standard iron solution (1 ml = 0·1 mg Fe) into 250-ml volume in a volumetric flask with distilled water and mix.

4.3 Preparation of Standard Iron Graph

4.3.1 Transfer 5·0, 10·0, 15·0, 20·0, 25·0 and 30·0 ml aliquot of standard iron solution (1 ml = 0·01 mg Fe) into 100-ml beakers. Bring the volume to approximately 20 ml with water. Place about 20 ml of water in a separate 100-ml beaker. This is blank and should be carried through all following steps along with the sample.

4.3.2 Set the beakers in a shallow water bath at 55 to 60°C. Add 2 ml of 10 percent hydroxylamine hydrochloride solution and stir. Allow the

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

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solutions to remain in the bath for 5 minutes.

4.3.3 Remove the beakers from the water bath. Add 0·5 ml of 0·1 percent 2 : 4 dinitrophenol indicator solution. Then, add 6 N ammonium hydroxide solution dropwise with stirring until the solution assumes a slight yellow colour. The *p*H is now about 4·4. Add very carefully 6 N HCl dropwise until the yellow colour of the solution is just discharged. The *p*H of the solution is about 2·6. Now add 1 ml of 1·5 percent *O*-phenanthroline solution. Stir well and cool to room temperature. The orange coloured iron, which is immediately produced, is due to the formation of the ferrous-*O*-phenanthroline complex.

4.3.4 Transfer to 50 ml volumetric flasks, dilute to mark with water and mix well. Allow the solutions to stand for a minimum of 15 minutes.

4.3.5 Transfer a suitable portion of the reagent blank solution to a 1 cm absorption cell and adjust the spectrophotometer to read 100 percent *T* with blank solution using a wavelength of 490 nm. While maintaining this adjustment, take readings of the calibrating solutions (*see* note).

NOTE — Colorimeters may be used instead of spectrophotometers for taking photometric readings. Optical filters having peak wave length of 470 nm and cuvette size 10 × 75 mm are suitable.

4.3.6 Plot the percent *T* values found for the corresponding milligrams of iron present on semi-logarithmic graph paper to obtain the 'Standard Fe graph'.

4.4 Procedure for Analysis of Coke (Both Calcined and Raw)

4.4.1 Weigh 5 to 25 g (generally 5 g) of dried coke, passing through 250-micron sieve, in a platinum crucible or dish and ash in a muffle furnace overnight at 620°C (*see* note). Remove the ash from furnace and cool in a desiccator.

NOTE — It is highly desirable to effect the ashing so that the ash may easily be removed from the container. At a temperature of 620°C, the ash from petroleum cokes have little or no tendency to fuse and stick to the crucible or dish. In all the ashing operations, filtered air (about 0·5 litre/minute) should be fed into the furnace to aid in the combustion of the carbon.

4.4.2 Carefully transfer the ash to a mortar. Add about 0·5 g of a 4 g portion of anhydrous Na_2CO_3 and grind lightly to a powder to ensure intimate contact of the ash with the flux. Transfer back the mixture to the platinum crucible, add the remainder of the 4 g portion of carbonate to the mortar, scour thoroughly and transfer to the crucible containing the ash. Mix the ash and the carbonate in the crucible thoroughly with a stirring rod. Place a platinum cover on the crucible, and fuse gently over a burner for 30 minutes. Swirl the melt about every 5 minutes during fusion to ensure complete digestion.

4.4.3 When fusion is complete, remove the crucible from the burner, swirl to distribute the melt on the sides of the crucible and allow to cool. Examine the cake, if it has a slight bluish-green colour, a slight amount of manganese is indicated. If the manganese is present, chlorine will be evolved on the addition HCl and will attack the platinum. To prevent this, moisten the coke with about 1 ml of ethanol, which will reduce the manganese iron and thus prevent such an attack.

4.4.4 Rinse off the outside of the platinum crucible with hot water, place the crucible and contents in a 250-ml beaker and add 25 ml of hot water. Cover the beaker with a watch glass and add slowly, from a burette, 6 N HCl to decompose the melt. When solution of the melt is complete, boil for several minutes on a hot plate and cool to room temperature. The solution, at this point, should be clear and free from any residue or precipitate.

4.4.5 Transfer the solution to a 100-ml volumetric flask, dilute to mark with water and mix well. This solution is now ready to be aliquoted for iron analysis.

4.4.6 Select a suitable aliquot containing not more than 0·3 mg Fe (and no more than 2 ppm of nickel which interferes in iron determination) for the colorimetric determination of iron. Since Ni greater than about 2 ppm is the chief interfering constitutent, the percent Ni in the coke should be determined before the Fe determination is made.

4.4.7 The data given in Table 1 is intended to assist in selecting the proper aliquot (of the 100 ml volume of the solution of the sample) for iron determination.

TABLE 1 SAMPLE SIZE

MASS OF COKE SAMPLJ, g (1)	EXPECTED PERCENT Fe IN COKE (2)	BEST ALIQUOT (3)
5	0·001	Use a large mass of sample
	0·01	20 ml
	0·05	5 ml
10	0·001	Use a large mass of sample
	0·01	10 ml
	0·05	5 ml
25	0·001	20 ml
	0·01	4 ml
	0·05	2 ml

4.4.8 Transfer by pipette the selected aliquot into a 100-ml beaker. Now, treat this solution exactly, as directed, for developing the orange red colour complex with *O*-phenanthroline, in the preparation of the standard graph (*see* **4.3.1** to **4.3.5**) and measure the percent transmittance of the solution. Using the percent transmittance thus found, read corresponding 'mg Fe' present in the aliquot of the sample taken from the 'Standard Fe Graph' prepared earlier.

4.4.9 Run a blank on all the reagents used. The 'blank' should be analyzed along with the sample and subjected to the same techniques and procedures. Deduct the 'mg' Fe found for the blank from the 'mg Fe' found for the sample to obtain the net 'mg' of Fe present in the aliquot sample.

5. CALCULATION

5.1 Let mg of Fe in aliquot of sample = N

$$\text{Total mg of Fe in 100 ml volume of sample} = N \times \frac{100}{\text{ml of aliquot taken}} = M$$

$$\text{Percent Fe (in coke spmple)} = \frac{(M \times 10^{-3}) \times 100}{\text{mass in g of coke sample taken for analysis}}$$

$$= \frac{0{\cdot}1 \times M}{\text{mass in g of coke sample taken for analysis}}$$

6. PRECISION

6.1 Results of duplicate tests shall not differ by more than the following:

Range of Iron Percent	*Repeatability*	*Reproducibility*
Under 0·05	0·01	0·02

Reprography Unit, BIS, New Delhi, India